IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

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Confirmation No.:	4484
Applicant:	HINTERMAN, *et al.*
Filed:	07/AUG/2006
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Examiner:	Kassa, Tigabu
Docket No.:	DC10010 PCT1
Customer No.:	00137
For:	Alkyl-Phenyl Silsesquioxane Resins Compositions

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

AFFIDAVIT UNDER 37 C.F.R. §1.132

Sir:

I, Gary Wieber, being duly sworn, say that:

1. I received a Ph.D degree in Chemistry from Colorado State University in Fort Collins, Colorado in 1990 and a Bachelor degree in Chemistry from The University of Michigan in Ann Arbor, MI in 1983.

2. I have been employed by the Dow Corning Corporation at Midland, Michigan since 1990, during which time I have been engaged in research and development activities in the fields of surface science, advanced ceramic materials and siloxane resins. I am a co-inventor of 12 U.S. patents and have authored/co-authored 5 papers and presentations at international conferences.

3. I am familiar with the above identified patent application.

4. Under my supervision, the following experiments were performed in an attempt to synthesize alkyl phenyl silsesquioxane resins according to above identified patent application.

Samples for examples were made at Dow Corning prior to March 13th 2003, the filing date of the Schlosser et al invention US2004/0180011A1.

The representative alkyl silsesquioxane resins of these examples are described using the M, D, T, and Q designation for the siloxy units present in the resin. The superscripts further describe the alkyl or phenyl substitute present on the siloxy unit. As used herein, Pr is $CH_3CH_2CH_2$--, Ph is C_6H_5--, and NH_2 is --$CH_2CH_2CH_2NH_2$. The subscripts describe the mole fraction of the siloxy unit in the resin. Thus, an alkyl-phenyl siloxane resin having a mole fraction of 0.50 for each siloxy unit is designated herein as $T^{Pr}_{0.50}\,T^{Ph}_{0.50}$.

Exhibit 1 corresponds to Example 1 of US2007/0132113A for a $T^{Pr}_{0.492}$-$T^{Ph}_{0.502}$ propyl phenyl silsesquioxane resin.
Exhibit 2 corresponds to Example 2 of US2007/0132113A for a $T^{Pr}_{0.692}$-$T^{Ph}_{0.306}$ propyl phenyl silsesquioxane resin.
Exhibit 3 corresponds to Example 3 of US2007/0132113A for a $T^{Pr}_{0.897}$-$T^{Ph}_{0.103}$ propyl phenyl silsesquioxane resin.
Exhibit 4 corresponds to Example 4 of US2007/0132113A for a $T^{Pr}_{0.46}$-$T^{Ph}_{0.45}$ - $D^{NH2}_{0.05}$ - $M_{0.03}$ alkyl phenyl silsesquioxane resin.
Exhibit 5 corresponds to Example 6 of US2007/0132113A for a $T^{Pr}_{0.32}$-$T^{Ph}_{0.31}$ - $D^{NH2}_{0.05}$ - $M_{0.33}$ alkyl phenyl silsesquioxane resin.

I declare that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true. I also declare that, at the time these statements were made, I knew that willful false statements and the like are punishable by a fine

or imprisonment, or both, under § 1001 of Title 18 of the United States Code, and that willful false statements may jeopardize the validity of the application, or any patent issuing from it.

Lanny Wiebe

Inventor Name

Date: June 7, 2010

Exhibit 1

DOW CORNING CORPORATION
MIDLAND MICHIGAN

LOCATION / BLDG. [redacted] PAGE [redacted]
FUNCTION
PROJECT NO.

SUBJECT T Propyl T Phenyl copolymer [redacted]

PROPRIETARY (*For legal protection each page must be dated, signed and any unused portion crossed out*)

$T^{Pr}_{0.50}T^{Ph}_{0.50}$

Lab book: [redacted]
Date Started: [redacted]

Reagents:	Targeted		Actual	
$PhSi(OMe)_3$	991.50g	5 mols	991.50g	[redacted]
$PrSi(OMe)_3$	821.40g	5 mols	821.60g	
FC24	0.56g	0.0037 mols	0.52g	
DI water	486.44g	27.00 mols	485.41g	
$CaCO_3$	3.47g	0.0347 mols	3.38g	
$MgSO_4$	4.00g		4.00g	

Theoretical Yield: 1121.89g with 100% condensation

Procedure:

- A 4 neck 3L round bottom flask was loaded with 991.50g of $PhSi(OMe)_3$ and 821.60g of $PrSi(OMe)_3$. The flask was equipped with an air driven teflon stir blade, thermometer, and a Dean Stark attached to a condenser.

JBH [redacted]

Time	Pot Temp(°C)		Comments
0:00	20.5		**Added 0.52g of FC24.** Target: 0.56g This amount is 0.05wt% of the theoretical yield.
4:00	20.5	*Exotherm*	**Added 323.26g of DI water.** Target: 324.29g = 18 mols This amount of water is 1.2x stoichiometrically needed to completely hydrolyze and condense the resin.
5:40	30		
7:00	60		
9:05	65		
11:05	65.5		**Turned heat on to distill out methanol.**
18:40	68		Methanol began to collect in Dean Stark.
3:29:30	75.5		Stopped heating. Total amount of alcohol removed: 778.33g Theoretical amount of alcohol that should be produced: 961.35g Percentage removed is: 81%
3:31:20	75		**Added 747.93g of toluene.** This amount of toluene would make the theoretical yield 60wt%.
3:33:00	56		**Began heating to distill out volatiles.**
3:45:40	70		Volatiles began to collect in Dean Stark.
4:09:30	75		Removed 115g of volatiles so far. At temperature the volatiles were one phase, but when cooled to room temperature there was two phases.
4:26:00	79		Stopped removing volatiles. Total amount removed: 200.7g
4:26:50	79		**Added 162.15g of DI water.** Target: 162.15g = 9 mols Kept on heating to azeotrope out water.
7:02:00	99		
7:04:00	100		Started to remove both top and bottom phases, because it appeared that methanol was coming over since water was separating out slow.
7:18:00	111		Stopped heating. Total amount of volatiles collected: 378.54g. Towards the end of removing water, water began to separate out slower. It was believed that more methanol was coming over. Therefore a few legs of the top phase were removed along with the water.
7:25:00	70		**Added 3.38g of $CaCO_3$** to neutralize the FC24. Targeted amount: 3.47g This is 10x the amount necessary to neutralize the FC24.

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Exhibit 1, cont.

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Stirred overnight at room temperature.

[redacted]

- Added 4g of $MgSO_4$ in case a small amount of water was present.
- Filtered through a Pyrex 350ml ASTM 40-60 C glass frit under vacuum using celite filter aid. Filtered through slowly. It filtered through hazy, but the $CaCO_3$ was filtered out.
- Because there was haze the resin was filtered through an Osmonics MAGNA Nylon Supported Plain 0.45um filter. Filtered through clear and colorless.

[redacted]

- Stripped on a rotovapor at 150-155°C at 0.4mm Hg for 1.5 hours.

Conclusions: Isolated Yield: 1075.44g Solventless resin was a clear colorless solid at room temperature and a liquid at 150°C. It had a slight amount of tackiness to it.

NVC 'A' : 150°C for 1 hour			
Pan wt. (g)	Resin soln. initial wt. (g)	Pan wt. + Resin soln. final wt. (g)	NVC
0.9899	1.5027	2.4900	99.83%
0.9868	1.5400	2.5259	99.94%
		Average:	99.89%

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[redacted]

Acid Number Titration Spreadsheet

JBH

[redacted]

Sample Information

[redacted]

	Run 1	Run 2
Sample Size (g)	3.4559	2.0209
Titrant Volume (mL)	0.0215	0.0135
NVC	100.00%	
Titrant Volume for Blank (mL)	0.0055	
Titrant conc. (N)	0.0130	

Calculations

	Run 1	Run 2	Average
Solution Acid #	0.00338	0.00289	0.00313
Solids Basis Acid #	0.00338	0.00289	0.00313
ppm HCl	2.2	1.9	2.0

*Acid number units: mg KOH/g

Comments:

Endpoint of titration determined by color change of Bromocresolpurople indicator in 50/50wt% Butanol/Toluene

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Exhibit 2

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FUNCTION _____
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SUBJECT T^{Pr} T^{Ph} copolymer

PROPRIETARY

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$T^{Pr}_{0.70}T^{Ph}_{0.30}$

Lab book
Date Started:

Reagents:	Targeted		Actual	
$PhSiCl_3$	291.30g	1.38 mols	291.70g	
$PrSiCl_3$	570.30g	3.21 mols	572.10g	
Toluene	483.63g		483.63g	
2-Propanol	213.65g		214.00g	
DI water	2136.52g	118.59mols	2136.52g	
Butyl Acetate	161.73g		161.60g	

Theoretical Yield: 483.63g with 100% condensation

Procedure:

- A 5L 4neck round bottom flask was loaded with 2,136.52g of DI water and 214.00g of 2-propanol. The flask was equipped with an air driven teflon stir blade, thermometer, and a condenser.
- A 2L Erlenmeyer flask was loaded with 483.63g of toluene, 291.70g of $PhSiCl_3$, and 572.10g of $PrSiCl_3$. These reagents were mixed together before they were added to the reaction. The amount of toluene added would make the theoretical yield 50wt% in toluene.
- The 5L flask was first heated to 70°C with a heating mantle.
- The reagents in the Erlenmeyer flask were then added using an addition funnel over a period of 16 minutes 40 seconds. The temperature was maintained between 74-78°C during addition by the use of an ice water bath and varying the addition rate.
- Once addition was done, the reaction was cooled slowly. The heating mantle was applied to slow the cooling rate, but no heat was applied.
- Transferred reaction into a 4L separatory funnel at 50°C.
- Removed water phase, which was on the bottom.
- Transferred reaction into a 3L 3neck round bottom flask and then azeotroped out the water. Dean Stark was not filled with toluene. Amount of water removed: 115.77g
- Did an azeotropic wash. This was done using 50.78g of DI water and 21.61g of 2-propanol. Targeted amount of water was 50.78g and the targeted amount of 2-propanol was 21.76g. The combined amount of water and IPA was 15wt% of the theoretical yield. The ratio of water to IPA was 70/30 wt%.
- Azeotroped out water. Amount collected: 59.44g Reaction was clear and colorless.
- Analyzed a 2.4g sample for acid content. Titration results were 148ppm HCl based on solution.
- Did another azeotropic wash. This was done using 50.78g of DI water and 21.61g of 2-propanol. Targeted amount of water was 50.78g and the targeted amount of 2-propanol was 21.76g.
- Azeotroped out water. Amount collected: 61.87g

- Stripped on a rotovapor at 125-130°C at 0.3mm Hg for 1 hour.

- Dissolved resin into butyl acetate. 161.73g of butyl acetate were added to 484.80g of stripped resin. The targeted amount of butyl acetate was 161.60g, which would make the resin 75wt%.

* Filtered through an Osmonics MAGNA Nylon supported plain 0.45 µm filter.

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Exhibit 2, cont.

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Conclusions: Solventless resin was a clear, colorless, highly viscous liquid at room temperature.
Isolated yield of solventless resin: 489.76g

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NVC for resin in butyl acetate

NVC 'A' : 150°C for 1 hour			
Pan wt. (g)	Resin soln. initial wt. (g)	Pan wt. + Resin soln. final wt. (g)	NVC
0.9864	1.9803	2.4707	74.95
0.9838	1.5671	2.1583	74.95
		Average:	74.95

*

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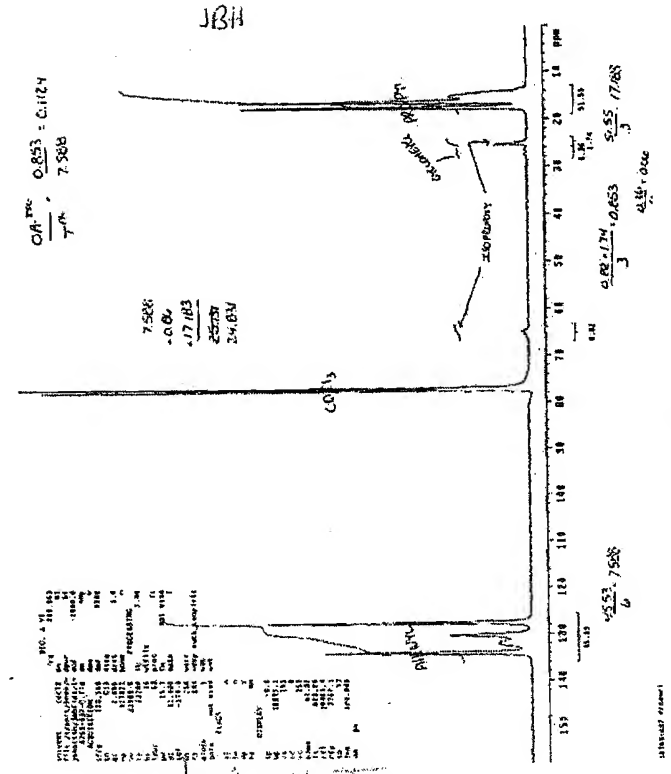

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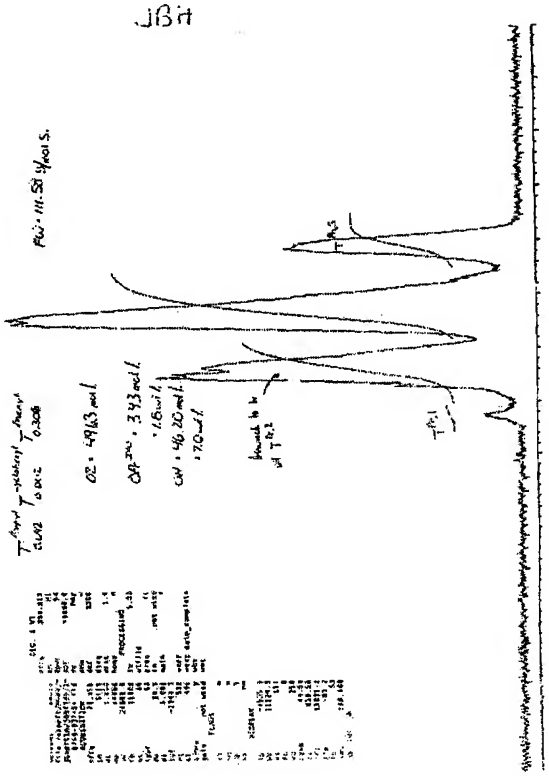

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Exhibit 3

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LOCATION / BLDG. _____ PAGE
FUNCTION _____
PROJECT NO. _____

SUBJECT T Propyl T Phenyl copolymer

PROPRIETARY (*For legal protection each page must be dated, signed and any unused portion crossed out*)

$T^{Pr}_{0.90}T^{Ph}_{0.10}$

Lab book
Date Started:

Reagents:	Targeted		Actual	
$PhSiCl_3$	103.60g	0.49 mols	103.76g	
$PrSiCl_3$	782.10g	4.41 mols	784.20g	
Toluene	482.54g		482.90g	
2-Propanol	227.88g		229.10g	
DI water	2278.82g		2278.82g	
Butyl Acetate	161.23g		161.09g	

Theoretical Yield: 482.54g with 100% condensation

Procedure:

- A 5L 4neck round bottom flask was loaded with 2,278.82g of DI water and 229.10g of 2-propanol. The flask was equipped with an air driven teflon stir blade, thermometer, and a condenser.
- A 2L Erlenmeyer flask was loaded with 182.90g of toluene, 103.76g of $PhSiCl_3$ and 784.20g of $PrSiCl_3$. These reagents were mixed together before they were added to the reaction. The amount of toluene added would make the theoretical yield 50wt% in toluene. JBH
- The 5L flask was first heated to 70°C with a heating mantle.
- The reagents in the Erlenmeyer flask were then added using an addition funnel over a period of 17 minutes 50 seconds. The temperature was maintained between 75-78°C during addition by the use of an ice water bath and varying the addition rate.
- Once addition was done, the reaction was cooled slowly. The heating mantle was applied to slow the cooling rate, but no heat was applied.
- Transferred reaction into a 4L separatory funnel at 50°C.
- Removed water phase, which was on the bottom.

- Transferred reaction into a 2L 3neck round bottom flask and then azeotroped out the water. Dean Stark was not filled with toluene. Amount of water collected: 3.62g
- Did an azeotropic wash. This was done using 50.67g of DI water and 21.75g of 2-propanol. Targeted amount of water was 50.67g and the targeted amount of 2-propanol was 21.71g. The combined amount of water and IPA was 15wt% of the theoretical yield. The ratio of water to IPA was 70/30 wt%.
- Azeotroped out water. Amount collected: 62.09g Reaction was clear and colorless.
- Analyzed a 2.4g sample for acid content. Titration results were 84ppm HCl based on solution.
- Did another azeotropic wash. This was done using 50.67g of DI water and 21.71g of 2-propanol. Azeotroped out water. Amount collected: 63.71g Resin was clear and colorless.
- Stripped on a rotovapor at 125-130°C at 0.3mm Hg for 1 hour.

- Dissolved resin into butyl acetate. 161.23g of butyl acetate were added to 483.27g of stripped resin. The targeted amount of butyl acetate was 161.09g, which would make the resin 75wt%.
- Filtered through an Osmonics MAGNA Nylon Supported Plain 0.45um filter. Filtered through clear and colorless.

Conclusions: Stripped resin was a clear, colorless, highly viscous liquid at room temperature.
Isolated yield of stripped resin: 488.82g

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Exhibit 3, cont.

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NVC of resin in butyl acetate

NVC 'A' : 150°C for 1 hour			
Pan wt. (g)	Resin soln. initial wt. (g)	Pan wt. + Resin soln. final wt. (g)	NVC
0.9899	1.7096	2.2574	74.14%
0.9849	1.5333	2.1236	74.26%
		Average:	74.20%

JBH

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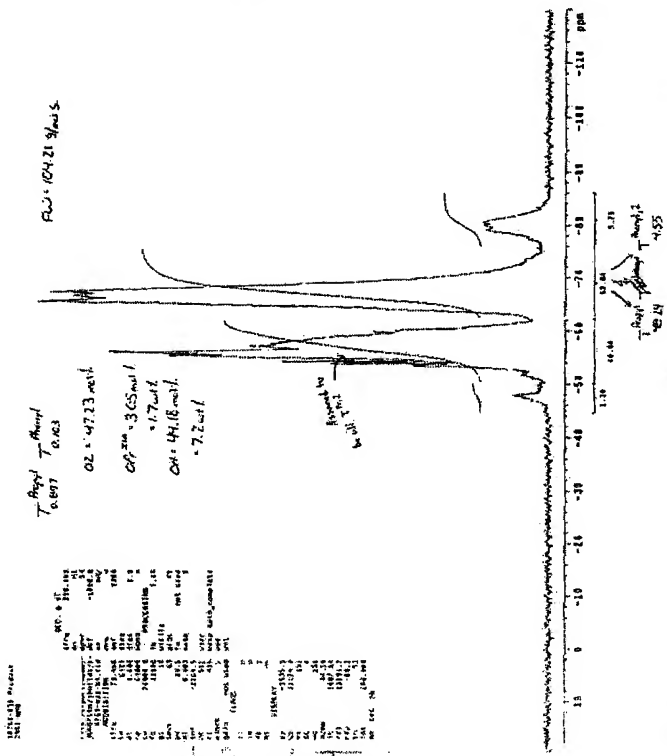

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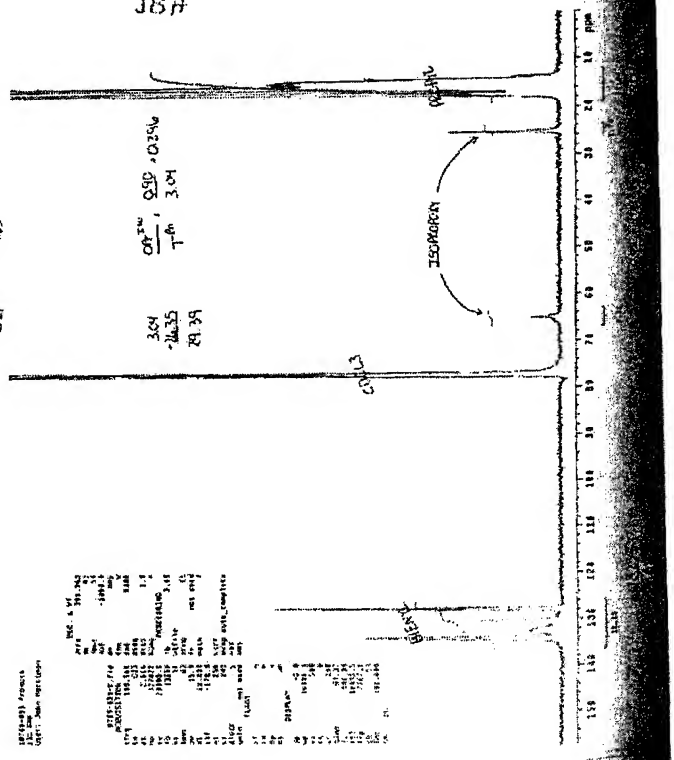

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LOCATION / BLDG. PAGE
FUNCTION

SUBJECT Solid $T^{Pr}T^{Ph}$ amine functional resin

PROPRIETARY (*For legal protection each page must be dated, signed and any unused portion crossed out*)

$M_{0.05}D^{NH2}{}_{0.05}T^{Pr}{}_{0.45}T^{Ph}{}_{0.45}$

Lab book
Date Started:

Reagents:	Targeted		Actual
$PhSi(OMe)_3$	446.15g	2.25 mols	446.24g
$PrSi(OMe)_3$	369.60g	2.25 mols	369.73g
$(OEt)_2MeSi(PrNH_2)$	47.85g	0.25 mols	47.85g
$EtOSiMe_3$	44.33g	0.375 mols	44.00g
Xylenes	376.38g		376.38g
DI water	194.24g	10.78 mols	194.26g
1.0N KOH (aq)	10.52g		10.55g
1.0N HCl (aq)	10.77g		10.77g

Theoretical Yield: 564.57g with 100% condensation including excess of $EtOSiMe_3$ that was added.

Procedure:

- A 3 neck 2L round bottom flask was loaded with 446.24g of $PhSi(OMe)_3$, 369.73g of $PrSi(OMe)_3$, 47.85g of $(OEt)_2MeSi(PrNH_2)$, and 376.38g of xylenes. . The flask was equipped with an air driven teflon stir blade, thermometer, and a condenser. Teflon was used for all glass joints and a teflon stir bearing was used for the glass stir rod.

Time	Pot Temp(°C)		Comments
0:00	21	*Exotherm*	**Added 116.00g of DI water. Target: 115.98g = 6.44 mols** Reaction turned opaque.
2:50	24		
10:11	30		
19:00	37		
21:30	39	▼	Reaction exotherm was probably not done, but the heat was turned on anyway to distill alcohol. Used a 12"vigreux column (insulated) with a horizontal condenser on top. Reaction still opaque.
38:50	63		Reaction was now clear.
3:49:30	130		Stopped heating. Normally the reaction would have been taken to 140°C, but due to the time, the reaction was stopped. Total amount of volatiles removed: 467.42g GC of volatiles: • 58.587% Methanol • 41.413% Xylenes
5:47:50	26		**Added 44.00g of $EtOSiMe_3$.** Target: 44.33g This is 1.5x the amount needed to arrive at the targeted amount of end cap.
5:49:00	25		**Added 25mL of xylenes** to make up for the volume of the Dean Stark.
5:52:35	25		**Added 78.26g of DI water along with 10.55g of 1.0N KOH (aq).** Targeted amount of water was 78.26g = 4.34 mols Targeted amount of 1.0N KOH(aq) was 10.52g Solid KOH is 0.1wt% based on theoretical yield. Began to heat to remove water via azeotrope. The vigreux column was replaced with a Dean Stark. Dean Stark was not filled with xylenes.
6:15:50	75		Volatiles began to collect in Dean Stark.
6:23:20	76		Removed 1 leg of the Dean Stark (top phase) because separation was slow. Aqueous phase was on the bottom. Immediately replaced what was removed with 25ml of xylenes. This helped the separation some.
7:54:00	110		Removed 1 leg of the Dean Stark because there was only one phase.

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		Pot temperature was not increasing. Immediately replaced what was removed with 25ml of xylenes.
8:11:00	136	One phase in Dean Stark Heated at reflux for 30 minutes.
8:40:30	135	Stopped heating. Total amount of volatiles removed during the azeotropic removal of water: 205.74g
8:47:00	50	Added 10.77g of 1.0N HCl (aq) Target: 10.77g This amount of HCl is 1.05x the amount needed to neutralize the KOH. Stirred overnight at room temperature.

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- Removed water via azeotrope. Total amount of aqueous phase removed: 12.44g
- Heated at reflux for 30 minutes after the majority of the water was removed.
- Pressure filtered through an Osmonics MAGNA Nylon Supported Plain 0.45um filter. Filtered through clear and colorless. Moderate filter rate.
- Stripped on a rotovapor at 155-160°C at 0.6mm Hg for 1 hour.

Conclusions: Isolated Yield: 529.94g Solventless resin was a clear and colorless solid at room temperature and a highly viscous liquid at 155°C. Total amount of water used in this reaction was **1.5x** the amount needed to completely hydrolyze and condense the resin.

Pages ___ to ___ read
and understood by
[signature]
on date ______

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Exhibit 5

PAGE DOW CORNING CORPORATION
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LOCATION / BLDG. _____
FUNCTION _____
PROJECT NO. _____

SUBJECT Liquid amino resin
PROPRIETARY

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$M_{0.30}D^{NH2}_{0.05}T^{Pr}_{0.325}T^{Ph}_{0.325}$

Lab book
Date Started:

Reagents:	Targeted		Actual
$PhSi(OMe)_3$	322.20g	1.625 mols	322.20g
$PrSi(OMe)_3$	266.95g	1.625 mols	266.95g
$(OEt)_2MeSi(PrNH_2)$	47.85g	0.25 mols	47.85g
Hexamethyldisiloxane	146.16g	1.80 mols Si	146.16g
FC24	0.27g		0.27g
DI water	230.83g	12.81 mols	230.84g
Toluene	360.05g		360.05g
1.0 N KOH	10.06g		10.08g
1.0 N HCl	10.29g		10.26g

Theoretical Yield: 540.07g with 100% condensation including excess of hexamethyldisiloxane that was added.

Procedure:

- A 3 neck 2L round bottom Indented Morton Type Flask was loaded with 322.20g of $PhSi(OMe)_3$ and 266.95g of $PrSi(OMe)_3$. The flask was equipped with an air driven teflon stir blade, thermometer, and a Dean Stark attached to a condenser.

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Time	Pot Temp(°C)		Comments
0:00	21		**Added 0.27g of FC24.** Target: 0.27g This is 0.05wt% of the theoretical yield.
2:00	21	*Exotherm*	**Added 67.11g of DI water.** Target: 67.11g = 3.725 mols
3:20	50		
5:00	54		
7:15	53	▼	Began to heat to **distill methanol.**
19:20	67		Volatiles began to collect in Dean Stark.
1:11:30	85		Stopped heating. Total amount of volatiles collected: 217.65g
1:16:30	75		**Added 146.16g of hexamethyldisiloxane.** This is 1.2x the amount needed to arrive at the targeted composition of 30 mol% endcap
1:19:10	61		**Added 16.24g of DI water.** Target: 16.21g = 0.90 mols Reaction turned hazy. Heated at 60°C for 4 hours. An additional hour was added compared to typical, because the reaction did not turn clear until after 3 hours
3:19:00	60		Still hazy.
4:15:00	60		Reaction is now clear.
5:21:00	59		**Added 47.85g of $(OEt)_2MeSi(PrNH_2)$.**
5:23:10	59		**Added 8.99g of DI water.** Target: 9.01g = 0.50 mols Reaction turned hazy.
5:24:00	60		Began to heat to **distill alcohol.**
5:32:00	68.5		Volatiles began to collect in Dean Stark.
5:37:30	70.5		Reaction is now clear.
6:11:10	95		Stopped heating. Total amount of volatiles removed: 88.06g GC of volatiles: • 70.305% Methanol • 25.483% Ethanol • 2.864% ? – Most likely Me_3SiOMe • 1.349% Hexamethyldisiloxane Reaction is clear and colorless.
6:14:00	94		**Added 360.05g of toluene.** This amount of toluene would make the theoretical yield 60wt% in toluene.

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Exhibit 5. cont.

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PROPRIETARY

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6:19:50	65	**Added 92.33g of DI water. Target: 92.33g = 5.12 mols** Began to heat to remove water via azeotrope. Dean Stark was not filled with toluene.
7:48:20	111	Majority of water removed.
8:00:00	113	Stopped heating. All water removed. Total amount of aqueous phase removed: 110.06g

Removed **sample 18769-070A** for ^{29}Si NMR analysis to determine if the OZ content was low enough to not require KOH bodying.

Results of NMR analysis of sample indicated that a fair amount of OZ was left. KOH bodying was therefore necessary.

Time	Pot Temp(°C)	Comments
0:00	22	**Added 10.08g of 1.0N KOH (aq) along with 46.17g of DI water.** Targeted amount of 1.0N KOH was 10.06g. This amount of solid KOH is 0.1wt% of the theoretical yield. Targeted amount of DI water was 46.17g = 2.56 mols Began to heat to remove water via azeotrope.
58:35	106	Removed 1 leg of the Dean Stark(~25ml). Did not replace with any toluene. JBil
1:04:40	111	Removed another leg.
1:08:30	115	Removed another leg.
1:13:30	117	Removed another leg.
1:18:10	118	Stopped heating.
2:05:00	35	**Added 10.26g of 1.0N HCl(aq) to neutralize the KOH.** Targeted amount of 1.0N HCl was 10.29g. This amount of HCl is 1.05x the amount needed to neutralize the KOH. Stirred at room temperature for 3 hours.
5:08:00	23	**Began to heat to remove water via azeotrope.**
5:37:20	117	Water removed. Stopped heating. Resin was clear except for some salt that was produced.

- Pressure filtered through an Osmonics MAGNA Nylon Supported Plain 0.45um filter. Filtered through clear and colorless.
- Stripped on a rotovapor at 120°C at 0.6mm Hg for 1 hour.

Conclusions: Isolated Yield: 525.75g Solventless resin a clear, colorless liquid at room temperature. The amount of incorporation of M in the final product was 95.6%. This was relative to the amount of EBB added with respect to T^{Ph} (initially added).

^{29}Si NMR integration data for sample 18769-070A

M	$D^{NH_2,1}$	$D^{NH_2,2}$	$T^{Pr,1}$	$T^{Pr,2}$	$T^{Ph,1}$	$T^{Ph,2}$	$T^{Ph,3}$
32.29	0.11	3.16	0.36	9.39	23.06	5.89	24.61

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